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WADC TECHNICAL REPORT 53-151
SUPPLEMENT 1

STUDY OF HARD COATING FOR ALUMINUM ALLOYS

F. J. Gillig

Cornell Aeronautical Laboratory, Inc.

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FOREWORD

This report was prepared by the Cornell Aeronautical Laboratory, Inc., under USAF Contract No. AF 18(600)-98. The contract was initiated under Research and Development Order No. 615-14, "Aluminum Alloys," and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. J. C. McGee acting as project engineer.

ABSTRACT

The program for the study of the effects of hard oxide coatings (produced by the MHC Process) on the properties of aluminum and its alloys was extended in order to provide additional data. The corrosion resistance in three environments was evaluated up to 11 months. The abrasion resistance showed another small decrease with the five-month additional exposure to atmospheric and high humidity conditions. Two treatments that were given the coatings on 61S and 75S alloys appear to alleviate the drastic reduction in fatigue strength brought about by the coatings. Attempts at retaining the abrasion resistance in a humid atmosphere were only moderately successful. Oil was found to have a detrimental effect on the resistance to an erosion type of wear.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. E. SORTE
Colonel, USAF
Chief, Materials Laboratory
Directorate of Research

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INTRODUCTION

The effects of hard oxide coatings (produced by the MHC Process) on the properties of aluminum and its alloys have been investigated in detail. The results of this investigation were summarized in WADC Technical Report 53-151.

In order to provide additional corrosion data for periods up to one year, the project contract date was extended from December 31, 1952 to May 31, 1953. During this period additional data were accrued for other properties of the coating and its effects on the base metal properties. The objectives of the latter test work were twofold: First, to improve the two objectionable features of the coating: its deleterious effect upon fatigue life and the loss of abrasion resistance with exposure to humidity, second, to provide additional data on fatigue life and high temperature properties.

The test program for this work has been outlined in previous reports. The results and data are presented in this report in the following sections.

TEST PROCEDURES AND RESULTS

Corrosion Resistance

Test specimens were exposed to three sets of conditions which are liable to lead to metallic corrosion:

1. Atmospheric exposure on an outdoor exposure rack on the roof on the Cornell Aeronautical Laboratory, Inc. This rack is shown in Figure 14 of WADC Technical Report 53-151.

2. High relative humidity at 80-90°F. These specimens were placed in desiccators containing distilled water and maintained at 80-90°F.

3. Exposure in a salt spray cabinet in accordance with A.S.T.M. Designation B117-49T.

The atmospheric exposure tests and the high humidity exposure tests showed no additional failures at the end of the 11-month period to those reported in WADC Technical Report 53-151 for the 7-month period. A few of the 24S bare alloy coatings failed in the atmospheric exposure tests. All specimens of this alloy failed at the end of 180 days in the humidity test. Some 24S Alclad and XA78S specimens also failed in this environment.

Additional failures were noted in the salt spray test. These failures are shown tabulated together with the previous results in Table I. With the exception of the 24S bare and Alclad wrought alloys, the coatings show excellent resistance to salt spray. The 220 cast alloy is not as good as the 356. However, when compared with regular anodized coatings and electroplated coatings, the hard oxide coatings are in general quite superior in the salt spray environment.

Abrasion Resistance

As explained in previous reports, the primary objective of the abrasion tests that were conducted was to determine the effect of exposure to atmospheric conditions and high humidity on the abrasion resistance of the hard oxide coatings. The results after exposures of 30, 60, and 90 days, and 6 months were reported in WADC Technical Report 53-151. The specimens were tested again at the end of the 11-month period and the results are given in Table II for the atmospheric exposure and Table III for the humidity exposure. If these values are plotted on Figures 15 through 21 of WADC Technical Report 53-151, they can be readily compared with all of the previous data. It can then be seen that, in both cases there was an additional decrease in abrasion resistance in the interval from 6 to 11 months. The decrease is not propor-

tional to the time and, in general, the values are only slightly below those for 6 months exposure.

Pull-Pull Fatigue Tests

The fatigue tests that were made during the main body of the evaluation program were all of the flexure type. It was felt that a comparison should be made between these data and some pull-pull tensile fatigue data. The possibility existed that due to the coating being at the point of highest stress in the flexure fatigue tests its effect might be overemphasized. The data plotted in Figure 1 show that this is not the case. The percentage reduction in fatigue strength when the coated and uncoated specimens are compared appears to be even greater in the case of the pull-pull fatigue tests.

Stress-Rupture Tests

During the course of the flame tests which were described in the Technical Report 53-151, it was noted that the oxide coating remained intact while the core metal became molten. It was stated that the coating would probably be of little value if heated under stress. Stress-rupture tests were run at 450°F on the 61S and 75S alloys to provide data on this point. The stresses were calculated on the basis of parent metal area remaining after coating. The data are given in Table IV. A peculiarity that was noted was that in the case of the coated specimens the reduction of area at the fracture was very limited while it was considerable in the uncoated specimens. This is undoubtedly due to the high compression strength of the coating at the temperature of the tests. The percent elongation was not affected noticeably.

Effect of Various Coating Treatments on the Abrasion Resistance of the Hard Coatings

A number of different treatments were applied to the coatings produced on 61S-T6 and 75S-T6 alloys in an attempt to prevent the loss of abrasion resistance of the hard coatings when exposed to a humid atmosphere.

These treatments were as follows:

1. Wax emulsion sealing - This treatment is a proprietary method of the Aluminum Company of America and consists essentially of impregnating the oxide film with wax in a colloidal solution.

2. Wax paste - The specimens were coated with Simoniz wax paste and the excess polished off. They were allowed to stand 48 hours before placing them in the humidity cabinet.

3. Zinc chromate - The specimens were sprayed with a film of zinc chromate primer just sufficient to form a continuous film over the surface.

4. Clear lacquer - This coating was applied in the same manner as the zinc chromate primer.

5. Lanolin base slushing oil - The specimens were dipped in a 20% solution of lanolin in a mineral solvent.

6. Hot oil - S.A.E. #30 oil was heated to 225°F and the specimens immersed for five minutes.

7. Chromate sealing - This treatment involved a 15-minute immersion in a 5% solution of potassium dichromate at 212°F.

8 & 9. The coated specimens were rinsed and transferred to 1% solutions of $BaCl_2$ and NH_4OH and made the cathode at sufficient voltage to just start the evolution of hydrogen - 10-12 volts.

The abrasion resistance of all coatings was measured at least 48 hours after they were applied. The specimens were then suspended in a humidity cabinet maintained at 190°F for a period of 20 days. The high temperature was used to accelerate the test, if possible, because of the relatively short time remaining before the end of the contract. The abrasion resistances were again measured upon removal from the humidity cabinet. These data are given in Table V.

Analysis of the data shows that all of the treatments cause a decrease in the abrasion resistance of the coatings before humidity exposure. The decrease was least in the case of those treatments not involving immersion in a hot water or oil solution. These treatments: wax paste, zinc chromate, and clear lacquer also resulted in the best abrasion resistance after exposure in the humidity cabinet.

It is interesting to note that both treatments involving the use of oils resulted in a very serious decrease in the abrasion resistance. Time did not permit further study of this point but it should be thoroughly investigated by those contemplating use of these coatings for increased abrasion resistance where they are liable to encounter similar conditions, such as, in hydraulic systems, gears, cams, etc.

The electrolysis treatments were aimed at reducing the concentration of sulphuric acid in the pores of the coating on the premise that it might be this residual acid that was contributing in a great measure to the reduction of abrasion resistance upon aging. It was thought that by making the coated specimen the cathode in a suitable electrolyte it might be possible to flush out the coating pores by evolving hydrogen and also neutralize the acid by precipitation of a weak basic or neutral compound.

This did not prove to be feasible as the hydrogen evolution took place preferentially at the microcracks in the coating. The abrasion resistance of specimens so treated fell off considerably after exposure in the humidity cabinet.

Effect of Treatments on Bending Fatigue

The problem of decreasing the serious loss of fatigue strength resulting from the coatings was attacked on the premises that softer coatings would be less likely to cause crack initiation in the parent metal and that numerous microcracks in the coating might result in more uniform work hardening of the surface rather than localized sites of low ductility.

In order to accomplish these results simultaneously, two methods were tried:

1. Treatment of the 61S and 75S alloys in hot 5% dichromate sealing solution which should also improve the corrosion resistance.

2. For the 61S alloy, coating the specimens in the solution treated condition and then aging them at 350°F for 10 hours.

The test results are shown plotted in Figures 2, 3, and 4 along with the results for coated and uncoated specimens that were determined previously. The chromate sealing treatment resulted in a definite increase in the fatigue strength at 10×10^6 cycles for both the alloys. The increase is not as great for the higher stresses in the case of the 61S alloy and it appears that the shape of the S-N curve has been altered. An increase in the 75S fatigue life is shown for all stress levels.

The aging treatment applied to the 61S coatings appears to have brought the endurance strength back to the level of the uncoated specimens. Unfortunately, time did not permit a redetermination of the S-N curve for uncoated reheat-treated specimens. The curve shown for the uncoated specimens is for the as-received material. It is possible that the reheat-treatment improved the base metal properties proportionately.

Although the treatments used above were aimed at producing a softer coating, they did not result in too great a loss in abrasion resistance as shown in Table VI.

CONCLUSIONS

The conclusions that are evident from the additional experimental work that was performed are as follows:

1. The coatings provide increased corrosion resistance for all of the aluminum alloys tested.

2. The alloy 24S does not appear to be suited to the hard coating process used.

3. The abrasion resistance continues to drop off after 11 months exposure to atmospheric and high humidity conditions.

4. The hard coatings do not affect the stress rupture strength of the 61S and 75S alloys at 450°F.

5. Suitable treatments for maintaining the abrasion resistance depend upon the exclusion of moisture.

6. The abrasion resistance of the coatings is reduced by the absorption of oil.

7. It is possible to alleviate the drastic effect that the coating has on the fatigue strength.

TABLE I

SALT SPRAY TEST DATA

Alloy	Days to Failure 0.0005 Inch Coating Thickness	0.001 Inch Coating Thickness	0.002 Inch Coating Thickness	0.003 Inch Coating Thickness	0.004 Inch Coating Thickness	0.005 Inch Coating Thickness
61S	220	*	*	330 (single pit) (one specimen)	*	*
XA78S	330 edge pits	*	*	*	300	330 pits starting
24S	60	90	90	150	180	150
24S Alclad	60	90	220	255	270	270
75S	220	*	*	300	330 pits at edge	330 pits at edge
356	**	330	**	330 pits starting	**	*
220	**	180	**	220	**	*

*Specimen has not failed in 330 days.

**The two casting alloys, 220 and 356 were tested with only 0.001, 0.003, and 0.005-inch coatings.

TABLE II

ABRASION TEST DATA FOR ELEVEN-MONTH EXPOSURE TO ATMOSPHERIC CONDITIONS

	Grams of 180 Mesh SiC Abrasive*					
Alloy	0.0005 Inch** Coating Thickness	0.001 Inch** Coating Thickness	0.002 Inch** Coating Thickness	0.003 Inch** Coating Thickness	0.004 Inch** Coating Thickness	0.005 Inch** Coating Thickness
61S	49	87	190	413	525	599
XA78S	79	120	236	266	389	465
24S	48	61	124	222	207	216
24S Alclad	26	122	248	546	557	420
75S	58	117	204	294	427	583
356	62	133	278	448	650	759
220	48	68	140	183	240	249

*All values given are in grams of 180 mesh SiC abrasive necessary to wear through coating when exposed to 20 mm. air pressure in an Arlt Abrasiometer.

**Coating thickness values are approximate -- actual values as given in previous report No. KB-802-M-5, Figures 15 through 21.

TABLE III

ABRASION TEST DATA FOR ELEVEN-MONTH EXPOSURE TO HIGH HUMIDITY 80-90°F

Alloy	Grams of 180 Mesh SiC Abrasive*					
	0.0005 Inch Coating Thickness	0.001 Inch Coating Thickness	0.002 Inch Coating Thickness	0.003 Inch Coating Thickness	0.004 Inch Coating Thickness	0.005 Inch Coating Thickness
61S	43	78	182	316	398	-
XA78S	49	85	144	181	278	289
24S	39	62	123	168	99	115
24S Alclad	23	124	241	410	423	580
75S	51	97	182	233	362	411
356	60	113	250	326	515	709
220	24	41	104	125	128	178

*All values given are in grams of 180 mesh SiC abrasive necessary to wear through coating when exposed to 20 mm. air pressure in an Arlt Abrasiometer.

TABLE IV

STRESS-RUPTURE TESTS

Alloy	Coating Thickness Inches	Test Temperature °F	*Stress P.S.I.	Time to Failure Hours	Elongation %	Stress to Rupture 100 Hours P.S.I.
75S-T6	0.000	450	10,000	135.5	23	10,800
"	0.000	"	12,500	25.5	18	
"	0.002	"	12,500	30.0	20	9,500
"	0.002	"	10,000	88.5	22	
"	0.004	"	10,000	106.0	22	10,100
"	0.004	"	12,500	30.0	O.G.	
61S-T6	0.000	"	14,000	106.0	10	14,100
"	0.000	"	16,000	35.25	12.5	
"	0.002	"	14,730	74.25	12.5	13,900
"	0.002	"	13,500	111.0	12.0	
"	0.004	"	14,000	95.0	10.0	13,800
"	0.004	"	16,000	45.0	16.0	

*Calculated on the basis of area of parent metal remaining after coating.

TABLE V

EFFECT OF VARIOUS COATING TREATMENTS ON THE ABRASION RESISTANCE

Alloy	Coating Thickness Inches	Treatment	Abrasion Resistance Before Humidity Exposure	Abrasion Resistance After Humidity Exposure*
61S-T6	0.003	None	593	436
"	"	**(1) wax emulsion	480	361
"	"	**(2) wax paste	584	465
"	"	**(3) zinc chromate	593	446
"	"	**(4) clear lacquer	562	477
"	"	**(5) lanolin base slushing oil	383	279
"	"	**(6) hot oil	313	294
"	"	**(7) chromate seal	543	381
75S-T6	"	None	462	348
"	"	wax emulsion	331	264
"	"	wax paste	442	392
"	"	zinc chromate	472	387
"	"	clear lacquer	426	342
"	"	lanolin base slushing oil	309	255
"	"	hot oil	244	212
"	"	chromate seal	390	305

TABLE V (Contd.)

EFFECT OF VARIOUS COATING TREATMENTS ON THE ABRASION RESISTANCE

Alloy	Coating Thickness Inches	Treatment	Abrasion Resistance	
			Before Humidity Exposure	After Humidity Exposure*
61S-T6	0.002	None	441	306
"	"	**(8) electrolysis in $BaCl_2$ - 2 min.	348	289
"	"	electrolysis in $BaCl_2$ - 10 min.	360	238
"	"	**(9) electrolysis in NH_4OH - 2 min.	397	280
"	"	electrolysis in NH_4OH-10 min.	360	255
75S-T6	"	None	420	339
"	"	**(8) electrolysis in $BaCl_2$ - 2 min.	318	222
"	"	electrolysis in $BaCl_2$ - 10 min.	120	108
"	"	**(9) electrolysis in NH_4OH - 2 min.	347	239
"	"	electrolysis in NH_4OH - 10 min.	279	238

*Exposed for 20 days in humidity cabinet at 190°F.

**Refer to text for description of coating treatments.

TABLE VI

ABRASION RESISTANCE OF SPECIMENS TREATED FOR INCREASED FATIGUE STRENGTH

Alloy	Coating Thickness Inches	Treatment	Abrasion Resistance* Before Treatment	After Treatment
61S-T6	0.001	Chromate Seal	194	135
"	0.003	" "	546	478
75S-T6	0.001	" "	119	116
"	0.003	" "	443	405
61S-T6	0.001	Aged 350°F - 10 hours	194	185
"	0.003	Aged 350°F - 10 hours	546	542

*Measured in grams of 180 mesh SiC necessary to wear through coating in an Arlt Abrasiometer operated at 20 mm. air pressure.

COMPARISON OF PULL—PULL & BENDING FATIGUE TEST DATA FOR

75S – T6 ALUMINUM — .051" THICK

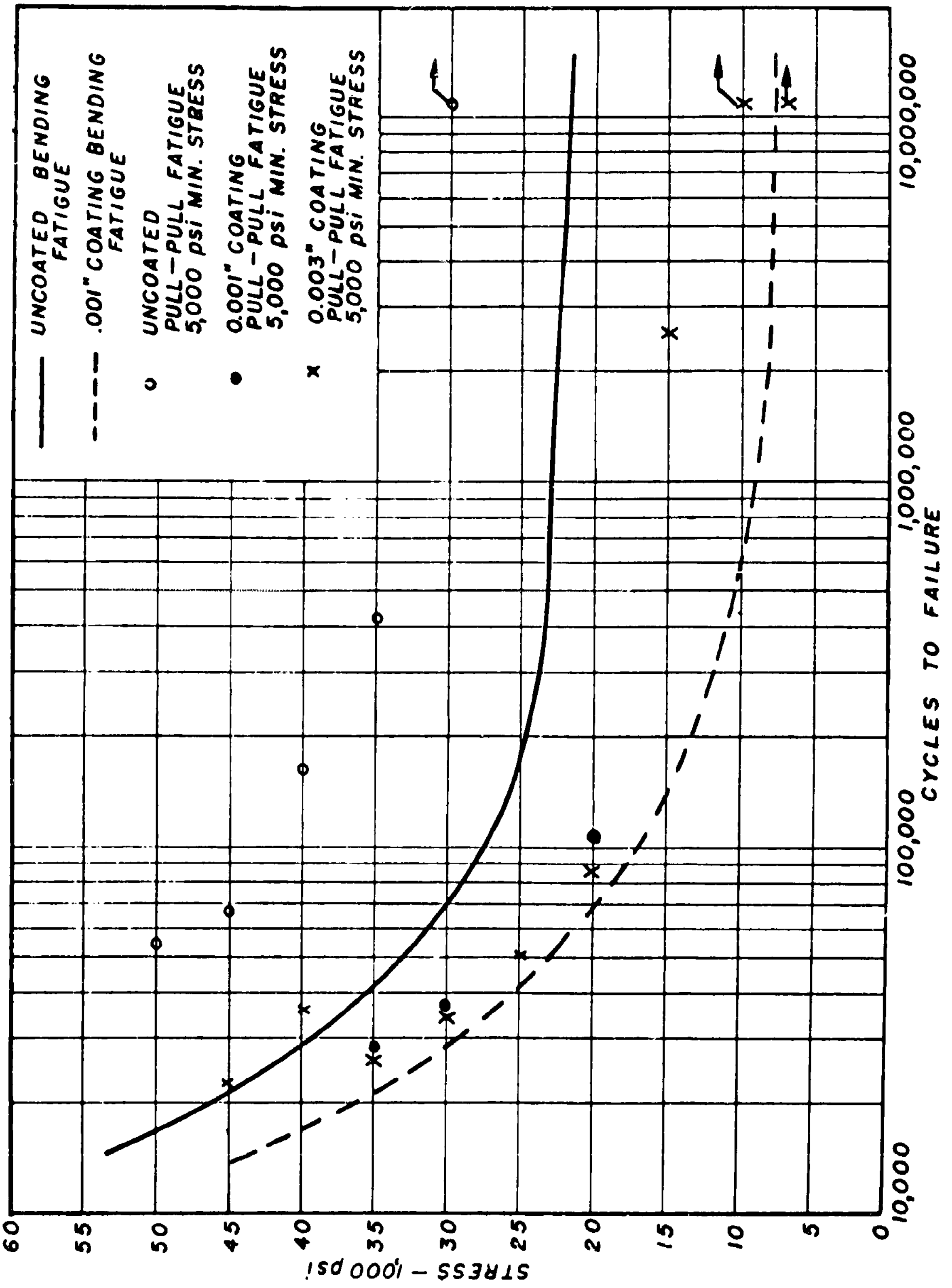

FIGURE 1

EFFECT OF CHROMATE SEALING TREATMENT
ON FATIGUE STRENGTH
OF HARD COATED 61 S-T6-.051" THICK

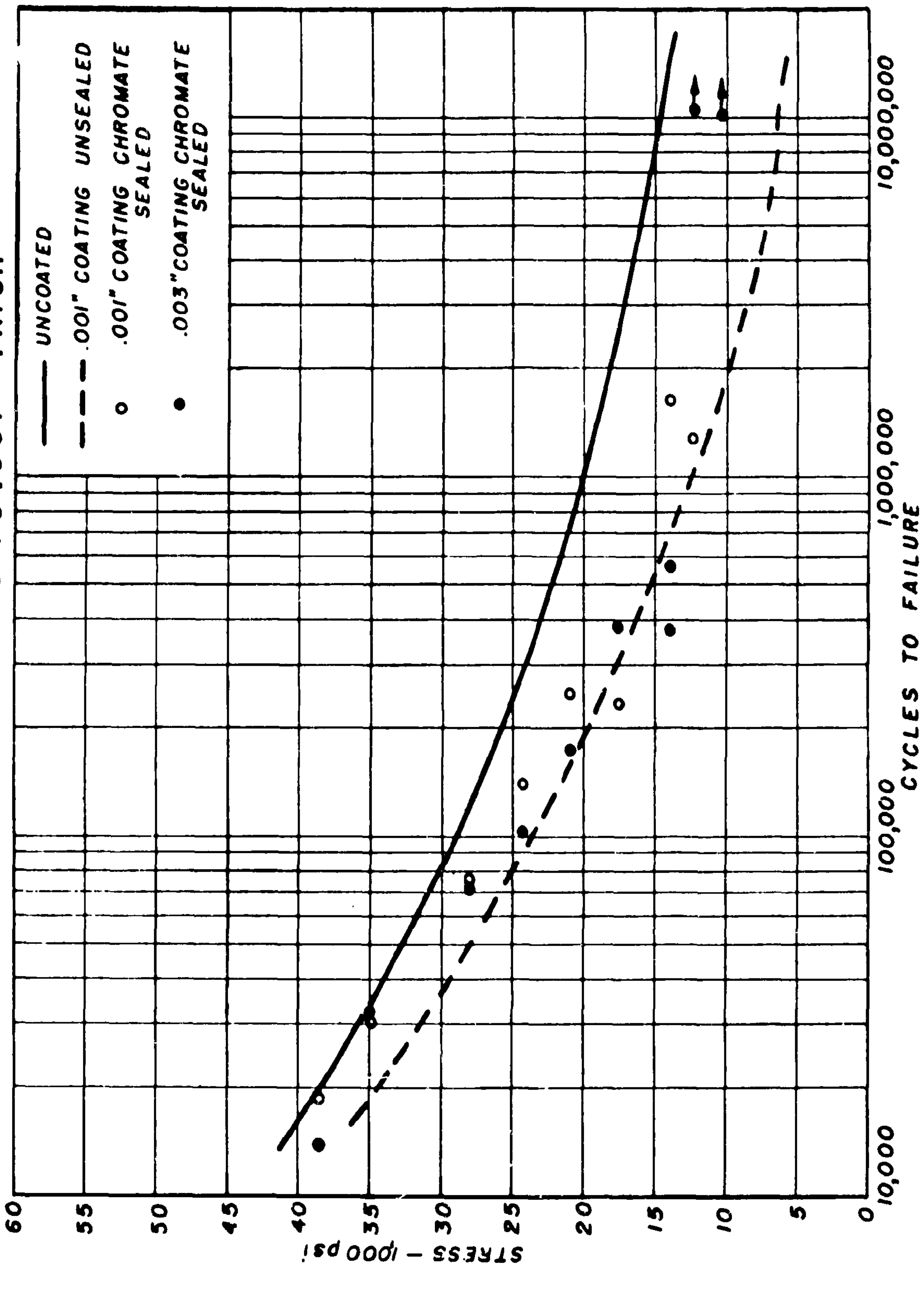

FIGURE 2

EFFECT OF CHROMATE SEALING TREATMENT
ON FATIGUE STRENGTH
OF HARD COATED 75S-T6-.051" THICK

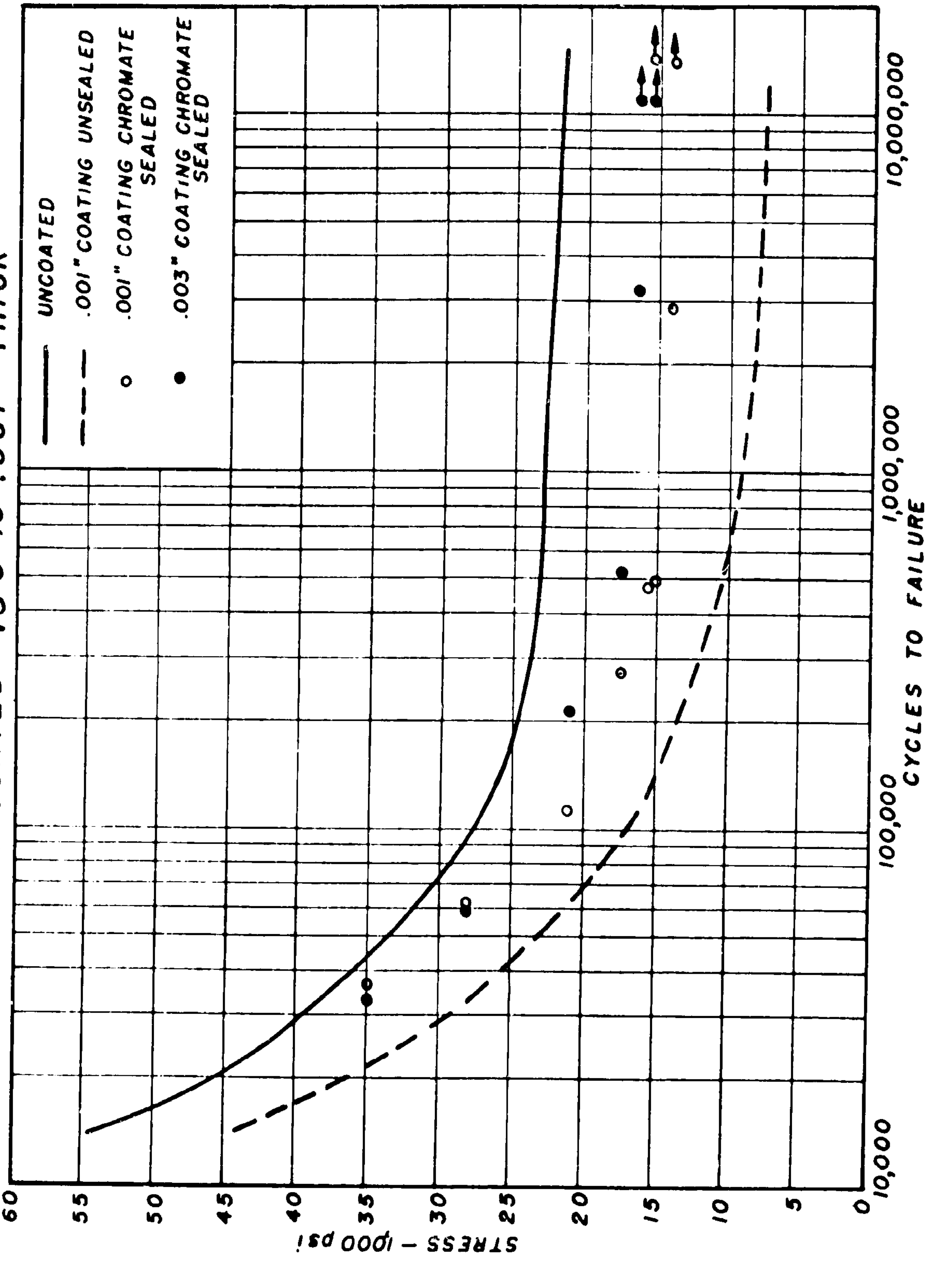

FIGURE 3

EFFECT OF 350°F AGING ON FATIGUE STRENGTH
OF HARD COATED 61 S -T6-.051" THICK

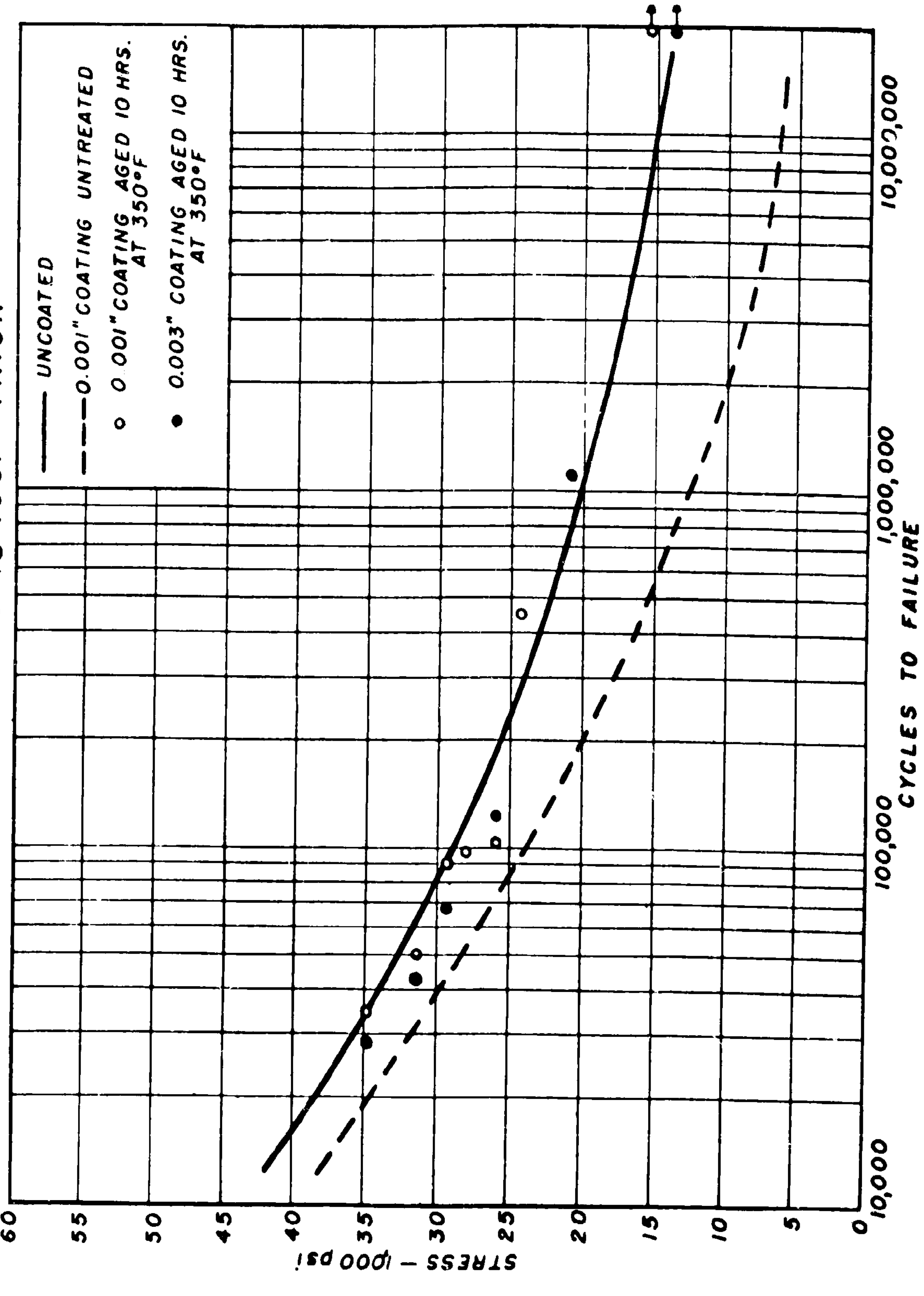

FIGURE 4